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IS 10745 (1983): Acetophenone [PCD 9: Organic Chemicals
Alcohols and Allied Products and Dye Intermediates]



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“Knowledge is such a treasure which cannot be stolen”

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IS 10745 - 1983

Indian Standard

**SPECIFICATION FOR
ACETOPHENONE**

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR ACETOPHENONE

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Indian Standard

SPECIFICATION FOR ACETOPHENONE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 8 December 1983, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Acetophenone is used in perfumery, in preparation of α chloroacetophenone (tear gas), in the manufacture of mandelic acid, phenylacetic acid and pharmaceuticals.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for acetophenone.

2. GRADES

2.1 There shall be two grades of the material, namely:

Grade 1 — intended for use in the manufacture of pharmaceuticals and perfumes, and

Grade 2 — intended for other industrial uses.

3. REQUIREMENTS

3.1 Description — The material shall be clear and almost colourless liquid.

*Rules for rounding off numerical values (revised),

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the method prescribed in Appendix A. Reference to the relevant clauses of the appendix is given in col 5 of Table 1.

TABLE 1 REQUIREMENTS FOR ACETOPHENONE

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Assay (as $C_6H_5 COCH_3$), percent by mass, <i>Min</i>	98.0	97.0	A-2
ii)	Acid value (as mg KOH/g), <i>Max</i>	0.2	0.5	A-3
iii)	Distillation range at 760 mmHg	196 to 204	194 to 204	A-4
iv)	Freezing point, °C, <i>Min</i>	18.0	17.0	A-5
v)	Relative density at 25°C/27°C	1.025 to 1.028	1.023 to 1.028	A-6
vi)	Halogen compounds	To pass the test	To pass the test	A-7

4. PACKING AND MARKING

4.1 **Packing** — The material may be packed in glass bottles, cans, drums, tank cars, tank trucks, etc.

4.2 The containers shall be securely closed and marked legibly and indelibly with the following information:

- Name and grade of the material;
- Mass of the material in the container;
- Name of the manufacturer and his recognized trade-mark, if any; and
- Lot or batch number in code or otherwise.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative test samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.2, and Table 1)

METHODS OF TEST FOR ACETOPHENONE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. ASSAY

A-2.1 Reagents

A-2.1.1 *Triethanolamine Aqueous Solution* — 0.5 N. Transfer 65 ml (74 g) of 98 percent triethanolamine to a 1 000-ml flask and dilute to make up with distilled water.

A-2.1.2 *Hydroxylamine Hydrochloride Alcoholic Solution* — 0.5 N. Dissolve 35 g of hydroxylamine hydrochloride in 150 ml of distilled water and dilute to 1 litre with isopropyl alcohol. Add 30 ml of 0.04 percent alcoholic solution of bromophenol blue indicator and from a burette add 0.5 N triethanolamine until the solution appears greenish blue by transmitted light. Prepare the fresh solution just before the analysis.

A-2.1.3 *Sulphuric Acid* — 0.5 N.

A-2.2 *Procedure* — Prepare a sufficient number of heat resistant pressure bottle to make all blank and sample determinations in duplicate. Replace the rubber gaskets if necessary and make sure the caps on the pressure bottles can be fastened securely. To each bottle add 65 ml of the neutralized hydroxylamine hydrochloride (A-2.1.2). From a burette add exactly 50 ml of the 0.5 N triethanolamine (A-2.1.1). Before capping, purge the bottles for 2 minutes with a gentle stream of nitrogen by means of a glass tube inserted through the neck of the bottle and clamped so that the opening is just above the surface of the liquid. Reserve two of the bottles for blanks.

*Specification for water for general laboratory use (second revision).

Into each of the other bottles introduce 1.5 to 1.8 g of sample by means of a suitable weighing pipette.

Enclose each bottle securely in a canvas bag and place the samples and blanks as close together as possible in a hot water bath maintained at $98 \pm 2^\circ\text{C}$ for 60 minutes. Remove the bottles from the bath and allow them to cool in air to room temperature. When the bottles have cooled, open the bags and then cool again slightly under tap water, and uncapped carefully to release the pressure. Allow the bottles to return to room temperature before titrating. Titrate each of the blanks with standard 0.5 N sulphuric acid (A-2.1.3) to a greenish blue end-point. Titrate each of the samples with sulphuric acid 0.5 N to the colour of the blanks, approaching the end-point dropwise until the colours match by transmitted light.

A-2.3 Calculation

$$\frac{(B-A) \times N \times 12.01}{M}$$

where

A = volume in ml of sulphuric acid required for the sample,

B = average volume in ml of sulphuric acid required for the blank,

N = normality of sulphuric acid, and

M = mass in g of sample taken.

A-3. DETERMINATION OF ACID VALUE

A-3.1 Reagents

A-3.1.1 Phenolphthalein Indicator — Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit (see IS : 323-1959*) and make the solution faintly pink by adding dilute sodium hydroxide solution.

A-3.1.2 Standard Potassium Hydroxide Solution — 0.02 N.

A-3.2 Procedure — Weigh 10 g of the sample into a 250-ml flask. Add 50 ml of rectified spirit (50 percent) which has been neutralized using 1 ml of phenolphthalein solution. Dissolve the contents completely. Titrate with standard potassium hydroxide solution shaking constantly until a pink colour which persists for 15 seconds is obtained.

*Specification for rectified spirit (revised).

A-3.3 Calculation

$$\text{Acid value, mg/g} = \frac{V \times N \times 0.056 \times 1\,000}{M}$$

where

V = volume in ml of standard potassium hydroxide solution,

N = normality of standard potassium hydroxide solution, and

M = mass in g of the material taken for the test.

A-4. DETERMINATION OF DISTILLATION RANGE

A-4.1 Carry out the determination of distillation range of the material according to the method prescribed in IS : 5298-1983*.

A-5. DETERMINATION OF CRYSTALLIZING POINT

A-5.0 Outline of the Method — The material is slowly cooled to determine its crystallizing point by observation of the temperature during crystallization under prescribed conditions.

A-5.1 Apparatus — The crystallizing point apparatus shall be as shown in Fig. 1. A thick glass tube of nominal size 100 × 25 mm is placed inside a large glass test-tube of nominal size 150 × 50 mm. The latter tube is flanged so that it may be supported centrally by the metal cover plate.

A-5.1.1 The wider tube is weighted with lead shots or similar material and the inner tube is closed by means of a cork, which carries a glass stirrer and through its centre a standard thermometer. The stirrer is a loop which surrounds the thermometer. The thermometer is so fixed in the cork that the thermometer's immersion mark is in level with the top of the cork and if the thermometer has a contraction chamber, the distance from the bottom of the bulb to the top of the contraction chamber shall not be more than 35 mm.

A-5.1.2 The wider tube is supported in a 1 000-ml tall-form beaker filled with cooling liquid to within 20 mm of the top. A thermometer for the cooling bath passes through a hold in the cover plate and is held by a rubber ring.

*Method for determination of distillation range and of distillation yield (first revision).

A-5.1.3 Thermometer — conforming to the Schedule Mark 22 of IS : 4825-1982* shall be used.

NOTE — Any thermometer of convenient range and similar requirements and accuracy may be used.

A-5.1.3.1 The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi or any other institution authorized by the Government of India to issue such a certificate.

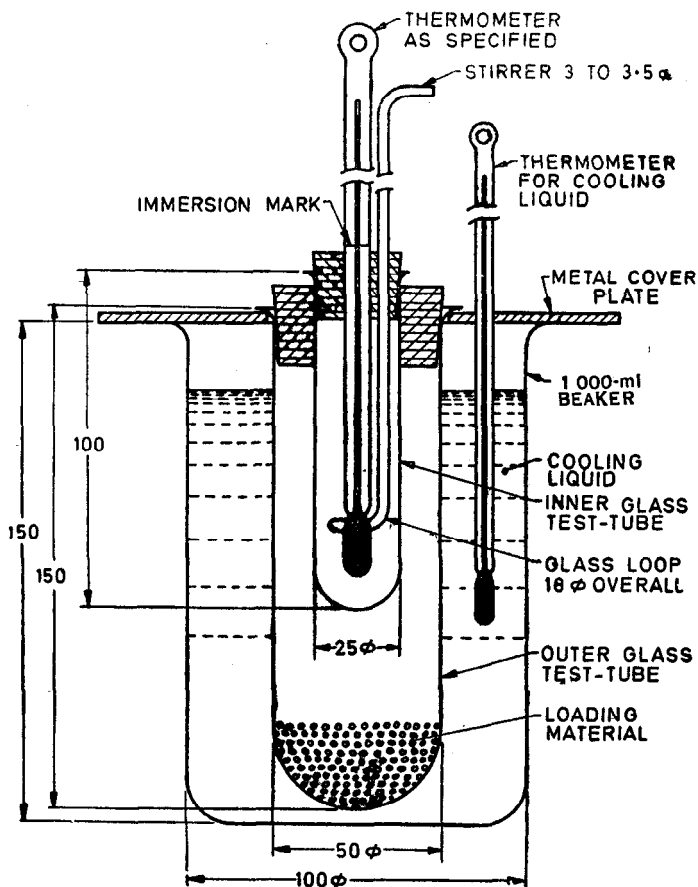


FIG. 1 APPARATUS FOR DETERMINATION OF CRYSTALLIZING POINT

*Specification for liquid-in-glass solid-stem reference thermometers (first revision).

A-5.2 Procedure — Take about 25 ml of the sample in the inner tube of the crystallizing point apparatus, immerse the thermometer and start stirring. Cool in water-bath adding ice to it to bring down the temperature. The temperature drop should be regulated to so that the rate of cooling is about 2°C per minute. When the temperature drops to about 20°C below the expected crystallization point, seeding crystal is introduced, if necessary, with stirring. At the point of crystallization, temperature will rise and then remain steady for the duration of crystallization. The steady temperature represents the crystallization point. Care should be taken to ensure that the apparatus is clean and dry before use.

A-6. DETERMINATION OF RELATIVE DENSITY

A-6.1 Apparatus

A-6.1.1 Pyknometer or Relative Density Bottle — 25 ml capacity.

A-6.1.2 Water-Bath — maintained at $27.0 \pm 0.2^\circ\text{C}$.

A-6.2 Procedure — Clean and dry the pyknometer or relative density bottle. Weigh it, fill it with freshly boiled distilled water, place it in the bath maintained at $27.0 \pm 0.2^\circ\text{C}$, and allow sufficient time (about 45 minutes) to attain the temperature of the bath. Then insert the capillary stopper which has also been brought to $27.0 \pm 0.2^\circ\text{C}$. Wipe the excess liquid from the stopper, remove the pyknometer or the relative density bottle from the bath, bring to room temperature and weigh. Empty the pyknometer or the relative density bottle, clean and dry it, and repeat the operation with the material at $27.0 \pm 0.2^\circ\text{C}$.

A-6.3 Calculation

$$\text{Relative density at } 27^\circ\text{C}/27^\circ\text{C} = \frac{A-B}{C-B}$$

where

A = mass in g of the pyknometer or relative density bottle filled with the material,

B = mass in g of the dry pyknometer or relative density bottle, and

C = mass in g of the pyknometer or relative density bottle filled with water.

A-7. TEST FOR HALOGEN COMPOUNDS

A-7.1 Wind a strip of 20 mesh copper gauze 15 mm wide and 50 mm long around the end of a copper wire. Heat the gauze in the non-luminous flame of a bunsen burner until it glows without colouring the flame green. Permit the gauze to cool and reheat several times until a good coat of oxide has formed. Apply 2 drops of the material with a medicine dropper to the cooled gauze, ignite and permit it to burn freely in the air. Again cool the gauze and add 2 more drops of the material and burn as before. This process is continued until a total of six drops have been added and ignited. Then hold the gauze in the outer edge of the bunsen flame, which had been adjusted to a height of about 40 mm.

A-7.1.1 The material shall be taken to have passed the test if not even a transient green colour is imparted to the flame.

A P P E N D I X B

(Clause 5.1)

SAMPLING PLAN FOR ACETOPHENONE**B-1. SCALE OF SAMPLING**

B-1.1 Lot — All the packages of the same grade and belonging to the same batch of manufacture shall be grouped together to constitute a lot.

B-1.2 For ascertaining the conformity of the material to the requirements of the specification, samples shall be tested from each lot separately.

B-1.3 The number of packages to be selected from a lot shall depend on the size of the lot and shall be according to Table 2.

TABLE 2 SCALE OF SAMPLING

NO. OF PACKAGES IN THE LOT	SAMPLE SIZE
(1)	(2)
Up to 25	2
26 „ 50	3
51 „ 100	5
101 „ 300	7
301 and above	10

B-1.3.1 These packages shall be selected at random from the lot. In order to ensure the randomness of selection, procedures given IS : 4905-1968*, may be followed.

B-2. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

B-2.1 From each of the packages selected according to **B-1.3**, a representative portion of the material shall be taken with the help of a suitable sampling instrument. The quantity of material to be taken for this purpose shall be adequate for making triplicate determination of all the requirements given in the specification.

B-2.2 Out of these portions, a small but approximately equal quantity of material shall be taken for each selected packages and mixed thoroughly so as to form a composite sample. The quantity of material in the composite sample shall be adequate for making triplicate determination for all the requirements to be tested on the composite sample (*see B-3*). The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-2.3 The remaining portion of the material from each selected package (after the quantity of material needed for the composite sample has been taken), shall be divided into three equal parts and transferred to separate bottles giving full identification particulars of samples on bottles. The material in each bottle shall constitute an individual sample. One set of bottles containing individual samples from all the selected packages shall be marked for the purchaser, another for the supplier and the third for the referee.

B-2.4 Referee Sample — The referee sample consisting of a composite sample (*see B-2.2*) and a set of individual samples (*see B-2.3*) shall bear the seals of both the purchaser and the supplier so as to be used in case of a dispute between the two.

B-3. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-3.1 Assay shall be done on each of the individual test samples (*see B-2.3*) and the remaining requirements given in Table 1 shall be tested on the composite sample (*see B-2.2*).

B-3.2 The lot shall be declared as conforming to the requirements of the specification if all the test results on the individual and composite samples satisfy the relevant specification requirements; otherwise not.

*Methods for random sampling.

(Continued from page 2)

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